



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 10 LABORATORY  
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**MEMORANDUM**

**TO:** Andy Smith, On Scene Coordinator  
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**cc:** Julie Wroble, Toxicologist  
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Risk Evaluation Unit

**FROM:** Jed Januch, Environmental Protection Specialist  
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Environmental Services Unit

**SUBJECT:** X-ray diffraction analysis of Burlington Hill mineral samples

**Project Code:** SFP-043A  
**Account Code:** 13T10P303DD210ZZLA00

The following pertains to the quality assurance (QA) documentation associated with analysis by x-ray diffraction (XRD) of three mineral samples submitted to the EPA Region 10 Laboratory. The samples are a subset of a larger group of samples previously analyzed by polarized light microscopy (PLM) and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). The samples originated from a sampling project conducted at the Burlington Hill site in Skagit County, Washington, on October 2, 2012. The goal of the XRD analysis was to identify the main mineral phases present and estimate the approximate concentration. Table 1 shows the identification numbers assigned to the samples and a description of the sample location.

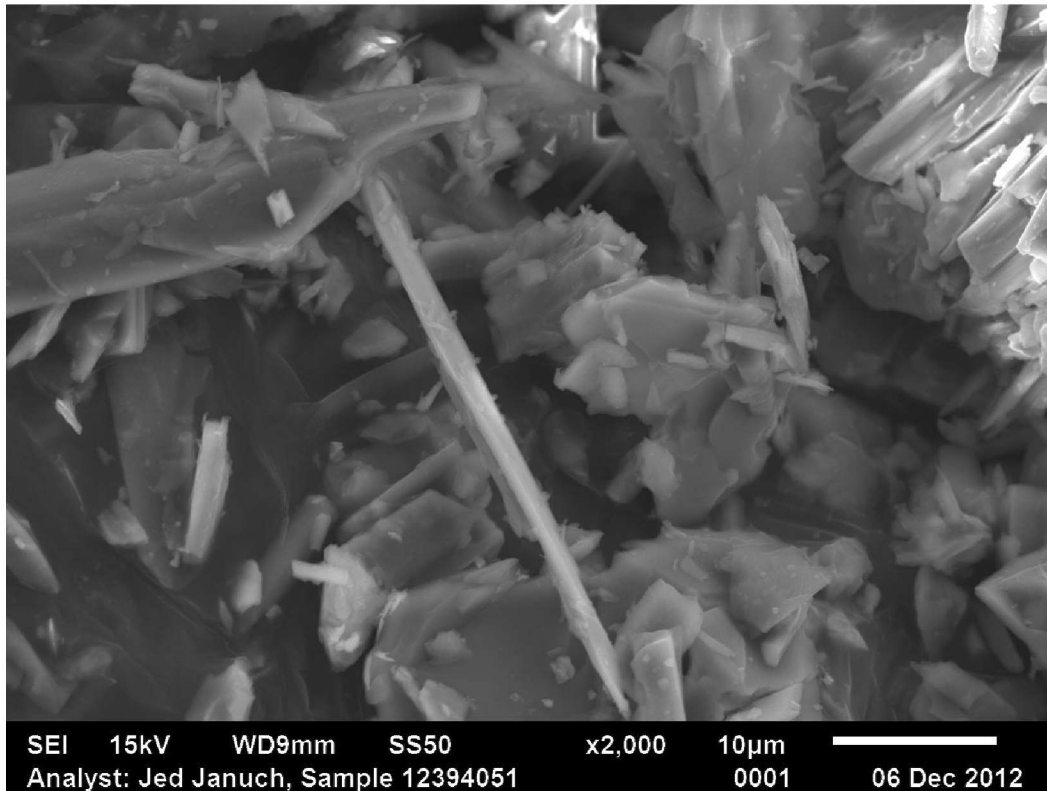
**Table 1**

<u>Field Identification Number</u>	<u>Laboratory Sample Number</u>	<u>Location Description</u>
12090101	12394051	Location 1- West
12090106	12394056	Location 2 - South of Driveway
12090110	12394060	Location 3 – North End Quarry

**METHODS**

I conducted the XRD analysis using Method XRD-QL for Compound Identification by X-ray Diffraction Analysis (U.S. EPA Manchester Laboratory) between November 5, 2012, and November 13, 2012. On January 8, 2013, the laboratory reported results of analysis by optical microscopy supplemented by examination of samples by scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). The XRD analysis provides verification of the presence of an amphibole (actinolite) in sample 12394051 based on internal crystalline structure, but does not distinguish the morphology (habit) of the mineral. A digital image that displays an example of an elongated actinolite structure from sample 12394051 is included in Figure 1.

**Figure 1-** Actinolite structures in sample 12394051 imaged with a JEOL 6510LV scanning electron microscope (SEM).



I used a Scintag X1 x-ray diffractometer to acquire diffraction data with Cobalt  $K\alpha$  radiation at a wavelength of 1.78897 angstroms ( $\text{\AA}$ ), generated at 36 mA and 45 kV. I recorded the XRD patterns at scan speeds of 0.5-15 degrees of two-theta ( $^{\circ}2\theta$ ) units per minute over a 2-76 degree range. Mineral phase identification was made by comparison with the Powder Diffraction File (PDF) maintained by the International Centre for Diffraction Data (ICDD, 2002).<sup>1</sup>

I prepared a specimen of each sample by light grinding with a corundum mortar and pestle. I packed a representative aliquot of each sample into a low background quartz dish for analysis by XRD.

Table 2 displays the qualitative abundance of each phase reported as major, minor, or trace amounts based on the intensity of diagnostic diffraction peaks and consideration of X-ray absorption characteristics. Corresponding numerical values are approximately greater than 20% by weight for major, 5-20% for minor, and less than 5% for trace amounts. The detection limit for XRD analysis is approximately 1-5%. Some, but not all, phases less than 5% of the bulk sample are included among the minerals present in trace amounts. I identified all major and minor phases in the samples and as many trace level phases as possible.

Quality control checks for the XRD analysis include the following:

1. The diffractometer's goniometer alignment was verified at the start and the end of the project (11/05/12 to 12/13/12) with Standard Reference Material SRM 1976, a flat plate of sintered alumina (corundum) provided by the National Institute of Standards and Technology (NIST).
2. At the beginning of each day of data collection, the alignment of the goniometer and stability of the X-ray intensity was checked by measuring the position and peak height of the 3.34 Å (101) peak of a novaculite (fine-grained quartz) reference plate.
3. The 3.34 Å peak of quartz was present in two of the samples and provided an internal reference for evaluation of sample displacement error.
4. National Bureau of Standards (NBS) standard reference material (SRM) 1867a (actinolite) was previously analyzed by XRD for other projects. The XRD pattern is suitable as a reference for this project.

## RESULTS

I detected a major concentration of actinolite in Sample 12394051 and other minerals including chlorite (clinochlore), mica (muscovite), and feldspar (albite). Previous analysis of Sample 12394051 by PLM and SEM/EDS confirms the presence of actinolite based on optical properties and chemistry by EDS. The XRD analysis provides verification of the actinolite identification based on crystal structure, but does not distinguish the habit or outward form of the mineral. In analysis of asbestos containing material, XRD data is complementary to the optical and analytical electron microscopy analyses, which provide information on the mineral habit. Therefore, not all actinolite in sample 12394051 identified by XRD is necessarily asbestos.

**Table 2** – Minerals identified by X-ray diffraction for samples from the Burlington Hill Site

### Mineral Identification by XRD, Project SFP-043A, Burlington Hill Site

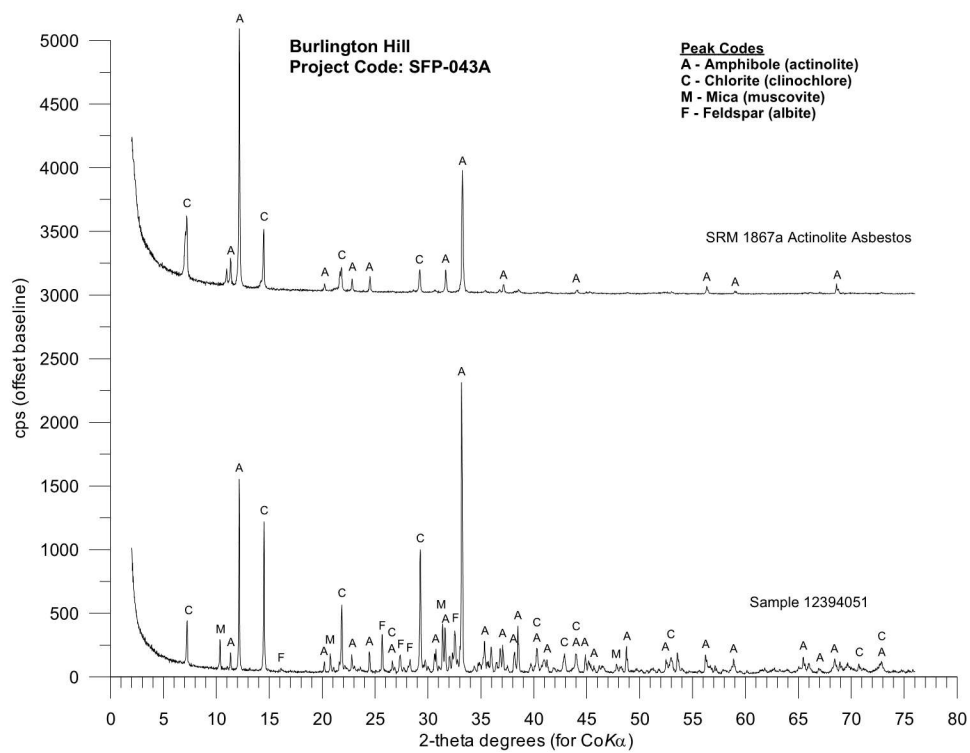
PHASE	IDEAL FORMULA	12394051	12394056	12394060
<u>SHEET SILICATES</u>				
Chlorite (clinochlore)	(Mg,Al,Fe)(Si <sub>6</sub> Al <sub>2</sub> O <sub>20</sub> (OH) <sub>16</sub>	M	M	M
Mica (muscovite)	K <sub>2</sub> Al <sub>4</sub> (Si <sub>6</sub> Al <sub>2</sub> O <sub>20</sub> ) <sub>4</sub> O <sub>10</sub> (OH,F) <sub>4</sub>	T	X	X
<u>FRAMEWORK SILICATES</u>				
Quartz	SiO <sub>2</sub>	ND	X	M
Feldspar (albite)	Na,Al,Si <sub>3</sub> O <sub>8</sub>	X	X	X
<u>CHAIN SILICATES</u>				
Amphibole (actinolite)	Ca <sub>2</sub> (Mg,Fe) <sub>5</sub> Si <sub>8</sub> O <sub>22</sub> (OH) <sub>2</sub>	M	ND	ND

#### NOTES

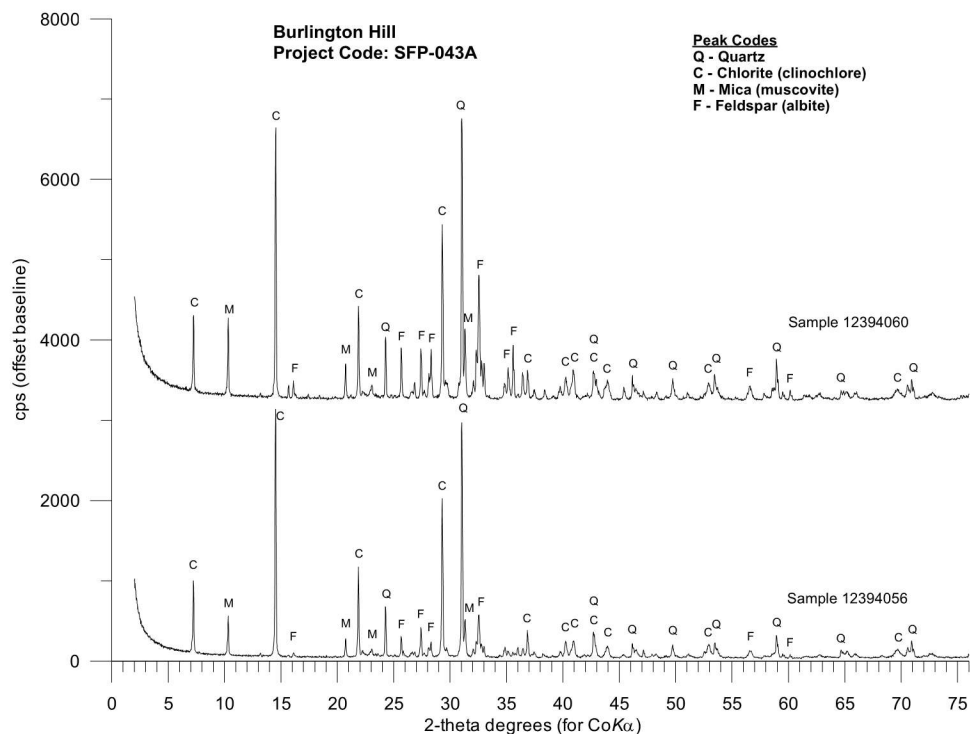
Qualitative abundance by peak area intensity designated by Major (M [>20%]), Minor (X [5-20%]), and Trace (T [<5%])  
 ND = None Detected

Actinolite was not detected by XRD or microscopy in samples 12394056 and 12394060. I did detect chlorite, mica, quartz, and feldspar in these samples.

**Figure 2** – XRD pattern of Sample 12394051 and NIST SRM 1867a Actinolite Asbestos. XRD analysis of Sample 12394051 was performed with the sample packed in a quartz dish (random orientation). The actinolite SRM was sedimented onto a silicon plate (preferred orientation) for XRD analysis.



**Figure 3**- XRD patterns for samples 12394056 and 12394060



## REFERENCES

- 1 ICDD, 2002, Powder diffraction file, release 2002: Newtown Square, Pennsylvania, International Centre for Diffraction Data, cd-rom.

ATTACHMENT – Data Documentation